भारतीय मानक **IS 5295 : 2023**

Indian Standard

इथाइलीन ग्लाइकॉल — विशिष्टि

(तीसरा पुनरीक्षण)

Ethylene Glycol — Specification

(Third Revision)

ICS 71.080.60

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July 2023 **Price Group 10**

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Ethylene glycol is also known as 1, 2 - ethanediol or more commonly as monoethylene glycol (MEG).

This standard was first published in 1969 and subsequently revised in 1985 and 2021. At the time of first revision, the committee decided to revise it in view of the growth in usage of the material in fibre and filament industry besides the general purpose use. The first revision covers two types of the material, namely, those catering to the need of the fibre and filament industry and those catering to the need of general purpose use such as antifreeze formulations, explosives and organic intermediates which are distinct from polyester fibres and filament industry. A separate specification for antifreeze coolant, namely IS 5759 was prepared by Lubricants and Related Products Sectional Committee which covers requirements and the methods of sampling and test for antifreeze formulations, consisting of mono ethylene glycol and additives such as corrosion inhibitors, foam suppressors and a dye required to provide corrosion and freeze protection to the engine cooling system and components.

In second revision, characteristic for both general grade and fibre grade were upgraded. Along with this, the requirement of desired purity, aldehyde as CH_3CHO or HCHO and clause for references were incorporated. Characteristics of ash along with its test method and colour after boiling for 4 h under total reflux were deleted. Further, marking clause was modified.

In this revision, the test methods have been revised, by taking considerably assistance from the following ASTM test methods:

a) ASTM E 394 Standard test method for iron in trace quantities using 1,10-phenanthroline method;
b) ASTM E 2313 Standard test method for aldehyde in mono-, di-, and triethylene glycol (using spectroscopy);
c) ASTM E 2409 Standard test method for glycol impurities in mono-, di-, tri and tetraethylene glycol and in mono- and dipropylene glycol (gas chromatographic method); and
d) ASTM E 2469 Standard test method for chloride in mono-, di-, and tri-ethylene glycol by ion chromatography.

The composition of the Committee, responsible for the formulation of this standard is given in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
ETHYLENE GLYCOL — SPECIFICATION
(Third Revision)

1 SCOPE

This standards prescribes requirements and the methods of sampling and test for ethylene glycol used in antifreeze formulations, explosives, organic intermediates and polyester fibres and filaments.

2 REFERENCES

The standards listed in Annex A contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards.

3 GRADE

There shall be two grades of the material, namely:

a) *General Grade* — 99 percent purity; and
b) *Fibre Grade* — 99.7 percent purity.

4 REQUIREMENTS

4.1 The material shall be colourless and free from suspended matter. It shall be miscible with water in all proportions at 20 °C.

4.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods given in col (5) and col (6) of Table 1.

4.2.1 *Quality of Reagents*

Unless specified otherwise, ‘pure chemicals’ and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

5 PRECAUTIONS IN HANDLING

The material shall be kept well protected from atmospheric humidity as it absorbs up to twice its mass of water vapour from the atmosphere.

6 PACKING AND MARKING

6.1 Packing

The material shall be packed in suitable and well closed containers with preferably, replaceable closure.

6.2 Marking

6.2.1 The containers shall be marked legibly and indelibly with the following information:

a) Name and grade of the material;
b) Name of manufacturer and his recognized trade-mark, if any;
c) Batch number;
d) Net mass of the material in the container;
e) Month and year of manufacture; and
f) Any other statutory requirements

6.2.2 For supplies of material in bulk, a test certificate containing the details mentioned at **6.2.1** shall be provided for each consignment.

6.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act,* 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

7 SAMPLING

The procedure for sampling and the criteria for conformity of the material shall be as prescribed in Annex C.

Table 1 Requirements for Ethylene Glycol

(*Clauses* 4.2, B-1.3.5, *and* B-7.2.5)

Sl No.	**Characteristic**	**Requirement**		**Method of Test, Ref to**	
		General Grade	Fibre Grade	Clause of Annex B	Indian Standard
(1)	(2)	(3)	(4)	(5)	(6)
i)	Colour, Pt-Co, *Max*	10	5	B-1	—
ii)	Relative density1) at 20 °C/20 °C	1.114 - 1.116	1.115 - 1.116	B-2	IS 1448 (Part 167)2)
iii)	Distillation range at 760 mmHg:				
	a) Initial boiling point, °C *Min*	194	195.5	—	IS 52983)
	b) The temperature at which 95 ml of material shall distil, °C, *Max*	200	199	—	
iv)	Moisture content, percent by mass, *Max*	0.2	0.1	B-32)	1448 (Part 175)/1448 (Part 182)
v)	Acidity (as acetic acid), ppm, *Max*	50	50	B-4	—
vi)	Ultra-Violet transmittance in 1 cm cell at			B-5	—
	a) 220 nm, *Min*	—	70 percent		—
	b) 275 nm, *Min*	—	90 percent		—
	c) 350 nm, *Min*	—	98 percent		—
vii)	Freezing point of equal volumes of material and water, *Max*	- 30 °C	—	B-6	—
viii)	Iron content, ppm, *Max*	—	0.1	B-7	—
ix)	Chlorides content (*as* Cl), ppm, *Max*	—	0.2	B-8	—
x)	Purity, percent, *Min*	99.0	99.7	B-9	—
xi)	Diethylene glycol content, ppm, *Max*	—	1 000	B-9	—
xii)	Aldehyde (*as* HCHO), ppm, *Max*	—	10	B-10	—

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1) Digital density meter may also be used for the determination of relative density.

2) In case of disputes, IS 1448 (Part 167) for determination of relative density and B-3 for determination of moisture content shall be the referee methods.

3) Distillation flask of 200 ml capacity shall be used instead of 125 ml as prescribed in IS 5298.

ANNEX A

(*Clause* 2)

LIST OF REFERRED STANDARDS

IS No.	*Title*
IS 264 : 2005	Nitric acid — Specification (*third revision*)
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
IS 915 : 2012/ ISO 1042 : 1998	Laboratory glassware — One-mark volumetric flasks (*third revision*)
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 1448 (Part 11) : 2004/ISO 3013 : 1997	Methods of test for petroleum and its products: Part 11 Petroleum products — Determination of freezing point of aviation fuels (*fourth revision*)
IS 1448 (Part 167) : 2018/ISO 12185 : 1996	Methods of test for petroleum and its products: Part 167 Determination of density — Oscillating u — Tube method
IS 1448 (Part 175) : 2020/ ISO 6296 : 2000	Methods of Test for Petroleum and its Products: Part 175 Petroleum products — Determination of water — Potentiometric Karl Fischer titration method
IS 1448 (Part 182) : 2020/ISO 12937 : 2000	Methods of Test for Petroleum and its Products: Part 182 Petroleum products — Determination of water — Coulometric Karl Fischer titration method
IS 2362 : 1993	Determination of water by Karl Fischer method — Test method (*second revision*)
IS 4161 : 2023	Nessler cylinders — Specification (*first revision*)
IS 5298 : 2013	Method for determination of distillation range and distillation yield (*second revision*)

ANNEX B

(*Table* 1)

METHOD OF TEST

B-1 DETERMINATION OF COLOUR

B-1.1 General

Two methods namely, Method A ‘Determination of colour by spectrophotometer’ and Method B ‘Determination of colour by visual method’ have been prescribed. In case of disputes, Method A shall be the referee method.

B-1.2 Method A

B-1.2.1 *Apparatus*

B-1.2.1.1 *Spectrophotometer*

B-1.2.1.2 *Erlenmeyer flask*, 1 000 ml, glass stopper

B-1.2.1.3 *Volumetric pipettes*, 1, 2 and 10 ml

B-1.2.1.4 *Cuvette*, 50 mm

B-1.2.2 *Reagents*

B-1.2.2.1 *Cobalt chloride* ($CoCl_2.6H_2O$)

B-1.2.2.2 *Potassium chloroplatinate* (K_2PtCl_6)

B-1.2.2.3 *Concentrated hydrochloric acid*, specific gravity of 1.19

B-1.2.2.4 *Platinum-Cobalt reference standards*

B-1.2.2.4.1 *Platinum-Cobalt stock solution*

Take 1.245 g potassium chloroplatinate, 1 g cobalt chloride, 500 ml distilled water and 100 ml concentrated hydrochloric acid in a 1 litre volumetric flask and shake it until dissolved. Dilute it up to the mark with distilled water. This solution has a colour of 500 platinum-cobalt scale units.

B-1.2.2.4.2 *Standard platinum — cobalt scale matching solutions*

To prepare the standard colour solutions of 5, 10 and 15 Pt-Co units, add 1 ml, 2 ml, and 3 ml of the 500 platinum-cobalt standard color solutions (*see* **B-1.2.2.4.1**) respectively to each of three 100 ml volumetric flask and dilute to the mark with distilled water. Cap the tubes and stored these standards for one year.

B-1.2.3 *Procedure*

Fill the clean and dry cuvette with distilled water and do blank correction. Introduce the sample into the sample holder and follow the instrument manufacturer directions. Then fill the cell with standard Pt-Co colour solution of 5, 10 and 15. Check the colour and it should give the same value.

B-1.2.4 *Precision*

Colour (Pt-Co units)	*Repeatability*	*Reproducibility*
25	3	10
75	5	15
165	7	22
265	10	31

B-1.3 Method B

B-1.3.1 *Apparatus*

B-1.3.1.1 *Nessler cylinder,* two, l00 ml capacity (*see* IS 4161)

B-1.3.1.2 *One-mark volumetric flasks,* 250 and 500 ml capacities (*see* IS 915)

B-1.3.2 *Reagents*

B-1.3.2.1 *Cobaltous chloride hexahydrate solid*

B-1.3.2.2 *Hydrochloric acid* (*see* IS 265)

B-1.3.2.3 *Chloroplatinic acid*

Dissolve 250 mg of platinum in small quantity of aqua regia contained in a glass or porcelain basin by heating on a water-bath. When the metal has dissolved, evaporate the solution to dryness. Add 1 ml of hydrochloric acid and again evaporate to dryness. Repeat this operation twice.

B-1.3.3 *Preparation of Colour Standard*

B-1.3.3.1 Dissolve 0.50 g of the cobaltous chloride hexahydrate and whole of the chloroplatinic acid in 50 ml of the hydrochloric acid. Warm, if necessary, to obtain a clear solution and after cooling, pour into the 500 ml one mark volumetric flask. Dilute with water to the mark.

B-1.3.3.2 Pipette 2.5 m1 and 5.0 ml of the solution (*see* **B-1.3.3.1**) into two separate 250 ml one-mark graduated volumetric flask and dilute with water to the mark. The dilute solutions correspond to a colour of 5 and 10 Pt-Co units respectively and should always be freshly prepared.

B-1.3.4 *Procedure*

Fill one of the Nessler cylinders to the mark with the material to be tested, and the other with the colour standard. Compare the colours using a white background.

B-1.3.5 *Result*

The material shall be taken to have passed the test if the colour of the test samples are not darker than that of the colour standard as required in Table 1.

B-2 DETERMINATION OF RELATIVE DENSITY

B-2.1 Apparatus

B-2.1.1 *Relative Density Bottle***,** 25 ml capacity

B-2.1.2 *Water-Bath***,** maintained at (20.0 ± 0.2) °C

B-2.1.3 *Thermometer***,** any convenient thermometer of a suitable range with 0.1 °C or 0.2 °C sub-divisions

B-2.2 Procedure

Clean and dry the relative density bottle, weigh and then fill with recently boiled and cooled water at 20 °C. Fill to overflowing by holding the relative density bottle on its stopper and immerse in the water-bath. Keep the entire bulb completely covered with water and hold at that temperature for 30 min. Carefully, remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry and weigh. Again clean and dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

B-2.3 Calculation

$$\text{Relative density at 20 °C/20 °C} = \frac{\mathrm{A-B}}{\mathrm{C-B}}$$

where

A = mass of the relative density bottle with the material, in g;

B = mass of the relative density bottle, in g; and

C = mass of the relative density bottle with water, in g.

B-3 DETERMINATION OF MOISTURE CONTENT

B-3.1 Procedure

Take about 10 g of the material, weigh accurately and determine the moisture content by the procedure as given in IS 2362.

B-4 DETERMINATION OF ACIDITY (*as* ACETIC ACID)

B-4.1 Outline of the Method

The material is titrated with standard sodium hydroxide solution using phenolphthalein as indicator.

B-4.2 Reagents

B-4.2.1 *Phenolphthalein Indicator*, 0.5 percent (*m*/ *v*)

Dissolve 0.5 g of phenolphthalein in 100 ml of rectified spirit (*see* IS 323) and make it faintly pink by the addition of dilute sodium hydroxide solution.

B-4.2.2 *Standard Sodium Hydroxide Solution*, 0.01 N

B-4.3 Procedure

Weigh accurately about 100 g of the material into a 500 ml conical flask to the nearest 0.1 g. Pour about 100 ml of freshly boiled and cooled distilled water and add 2 drops to 3 drops of phenolphthalein indicator. Mix well and note the colour of the solution. A pink colouration indicates the presence of alkalinity. If the solution is colourless, titrate with standard sodium hydroxide solution until a pale pink colour persists for more than 15 s. Take 100 ml of distilled water in another 500 ml conical flask and carry out blank test.

B-4.4 Calculation

Acidity (*as* acetic acid), percent by mass =

$$\frac{(V_1 - V_2) \times N \times 0.06}{M} \times 100$$

where

V_1 = volume of standard sodium hydroxide solution used in the test with the material, in ml;

V_2 = volume of standard sodium hydroxide solution used in blank test, in ml;

N = normality of standard sodium hydroxide solution; and

M = mass of the material taken for the test, in g.

B-5 DETERMINATION OF UV TRANSMITTANCE

B-5.1 General

The UV transmittance of ethylene glycol is a measure of purity of the product. The transmittance of a sample is measured at 220 nm, 275 nm and 350 nm on the UV-spectrophotometer. Low transmission at any wave length indicates low purity with the wave length being characteristic of the impurity.

B-5.2 Apparatus

B-5.2.1 *Ultraviolet Spectrophotometer*

B-5.2.2 *Ultra-violet Light Source*

B-5.2.3 *Silica Cells,* 1 cm

B-5.3 Procedure

Inspect all equipment to ensure operability and the cells to insure that they are absolutely clean and dry. Prepare the spectrophotometer for use with the ultraviolet light source. Adjust transmission to 100 percent with distilled water in cell (against distilled water reference for double beam operation) at 220 nm. Fill the silica cell (insuring cell is dry before use) with ethylene glycol and the reference cell with distilled water. Place the cells in the cell holder and the cell holder in spectrophotometer. Determine the transmittance of the sample at 220 nm, 275 nm and 350 nm.

B-5.4 Report

Report results as percent transmission of virgin ethylene glycol at 220 nm, 275 nm and 350 nm.

B-6 DETERMINATION OF FREEZING POINT

B-6.1 Preparation of Test Sample

Mix thoroughly equal volumes of the material and water.

B-6.2 Procedure

Determine the freezing point of the test sample by following Method B of IS 1448 (Part 11), except that the sample shall not be dried before determining the freezing.

B-7 DETERMINATION OF IRON CONTENT

B-7.1 General

Two methods, namely Method A for 'Determination of iron content by using thioglycolic acid' and Method B for 'Determination of iron content by using 1, 10-phenonthroline' have been prescribed. In case of dispute Method B shall be the referee method.

B-7.2 Method A

B-7.2.1 *Outline of the Method*

In alkaline medium, thioglycolic acid gives a reddish violet solution with ferrous and ferric iron. In this method the material is treated with thioglycolic acid and ammonia and the colour produced compared with that produced by standard iron solution under identical conditions.

B-7.2.2 *Apparatus*

B-7.2.2.1 *Nessler cylinders,* l00 ml capacity (*see* IS 4161)

B-7.2.3 *Reagents*

B-7.2.3.1 *Thioglycolic acid solution*

Dissolve 10 g of thioglycolic acid in distilled water to make 100 ml solution.

B-7.2.3.2 *Concentrated ammonia*, of 0.90 relative density

B-7.2.3.3 *Standard iron solution*

Weigh 0.702 g of ferrous ammonium sulphate and dissolve in 100 ml of water. Make up 1 ml of this solution to 100 ml with water. 1 ml of this solution then contains 0.01 mg of iron.

B-7.2.4 *Procedure*

Weigh accurately 50 g of the material into a nessler cylinder, pour 25 ml of water, add 2.5 ml of thioglycolic acid solution and 5 ml of concentrated ammonia. Mix thoroughly and make up the volume to 100 ml with water. Take, in another nessler cylinder, 75 ml distilled water and 1 ml of standard iron solution, add 2.5 ml of thioglycolic acid solution and 5 ml of concentrated ammonia. Mix thoroughly, make up the volume to 100 ml with water and after 10 min compare the colours of both solutions.

B-7.2.5 The iron content of the material shall be regarded as not having exceeded the limit prescribed in Table 1, if the intensity of colour produced with the material is not deeper than that produced with standard iron solution.

B-7.3 Method B

B-7.3.1 *Outline of the Method*

This test method is based upon a photometric determination of the 1,10-phenanthroline complex with the iron (II) ion. The sample is dissolved in a suitable solvent and the iron is reduced to the divalent state by the addition of hydroxylamine hydrochloride. The colour is then developed, by the addition of 1,10-phenanthroline. After a short reaction period, the absorbance of the solution is measured at 510 nm using a suitable photometer.

B-7.3.2 *Apparatus*

B-7.3.2.1 *UV-Vis spectrophotometer*

B-7.3.2.2 *Cuvette*, 10 mm or 50 mm

B-7.3.3 *Reagents*

B-7.3.3.1 *Hydrochloric acid*

B-7.3.3.2 *Hydroxylamine hydrochloride*, 10 percent

Dissolve 10 g of hydroxylamine hydrochloride in 100 ml of water.

B-7.3.3.3 *Ammonium acetate – acetic acid solution*

Dissolve 100 g ammonium acetate ($NH_4C_2H_3O_2$) in 600 ml water and filter. Add 200 ml of glacial acetic acid, dilute to 1 litre with water.

B-7.3.3.4 *1,10-Phenonthroline solution*, 0.3 percent

Dissolve 0.3 g of 1, 10-phenonthroline in 10 ml of methanol and dilute to 100 ml with distilled water.

B-7.3.3.5 *Iron standard solution*, certified reference material, 1 000 mg/kg.

B-7.3.4 *Calibration*

Prepare 100 mg/kg and then 10 mg/kg iron solution by diluting certified reference material of iron solution. From 10 mg/kg iron solution, take 0.5 ml, 1 ml, 2 ml, 3 ml, 4 ml, 5 ml and 6 ml iron solution (containing 5 µg, 10 µg, 20 µg, 30 µg, 40 µg, 60 µg of iron respectively) in 100 ml volumetric flask and follow the procedure as given at **B-7.3.5.1** for colour development.

Measure the absorbance of each calibration standard at 510 nm using 10 mm cell. Now plot the standard curve with µg of iron on the x-axis and absorbance on y-axis.

B-7.3.5 *Procedure*

B-7.3.5.1 Mix the sample properly and transfer 80 ml of it in a beaker. To it add 2.0 ml of hydroxylamine hydrochloride solution. Add stopper and homogenize the solution by swirling the flask. Now add 5 ml of 1,10-phenanthroline solution. Check *p*H and adjust it between 3 *p*H- 4 *p*H, if required by ammonium acetate buffer solution. Add 5 ml of ammonium acetate – acetic acid solution and transfer it to 100 ml volumetric flask. Dilute it to 100 ml with distilled water. Again stopper and homogenize the solution by swirling the flask. Allow the solution to stand at room temperature for around 15 min for development of colour. After 15 min measure the absorbance at wavelength of 510 nm.

B-7.3.5.2 Similarly, follow the procedure as given at **B-7.3.5.1** for colour development in distilled water which is used as blank to set the instrument at zero absorbance.

B-7.3.6 *Calculation*

$$\text{Iron, mg/kg} = \frac{\text{Iron from calibration graph, in µg}}{\text{Volume of sample} \times \text{Density of sample}}$$

B-7.3.7 *Results*

Report the iron content to the nearest 0.01 mg/kg.

NOTE — 1 ppm = 1 mg/kg

B-8 DETERMINATION OF CHLORIDES (*as* Cl)

B-8.1 General

Three test methods, namely Method A ‘Determination of chloride by turbidity method’, Method B ‘Determination of chloride by microprocessor-based potentiometric auto titrator’ and Method C ‘Determination of chloride by ion-chromatography’ have been prescribed for the determination of chlorides in ethylene glycol by three different techniques. In case of disputes, ion-chromatography (Method C) shall be the referee method.

B-8.2 Method A

B-8.2.1 *Outline of the Method*

Chloride ion in glycol is precipitated as silver chloride by the addition of silver nitrate solution to an acidic solution of the glycol. The resulting turbidity is compared in an equivalent volume of solutions as a measure of the total halides in the range of 0.0 ppm to 0.5 ppm.

B-8.2.2 *Apparatus*

B-8.2.2.1 *Turbidity meter*

B-8.2.2.2 *Turbidity cell*

B-8.2.2.3 *Nessler tube,* capacity 100 ml

B-8.2.2.4 *Volumetric flask*

B-8.2.3 *Reagents*

B-8.2.3.1 *Nitric acid*, dilute, 1 : 1 dilution with chloride free distilled water

B-8.2.3.2 *Silver nitrate*, 1 percent solution in chloride free distilled water

B-8.2.3.3 *Sodium chloride*

B-8.2.3.4 *Nitric acid* (*see* IS 264)

B-8.2.3.5 *Chloride free water*, ultra-pure water, or triple distilled water

B-8.2.4 *Chloride Standard Preparation*

B-8.2.4.1 *Chloride standard solution* (1 ml = 5.58 µg chloride ion)

Accurately weigh 0.920 g of sodium chloride and dissolve with water in 100 ml volumetric flask. Make it up to 100 ml with water and mix thoroughly. Transfer 10 ml of aliquot into another 100 ml volumetric flask, dilute it up to 100 ml mark with distilled water mix thoroughly. Further, from this solution take 10 ml aliquot in 1 litre volumetric flask. Make it up to mark and mix thoroughly. This final solution contains 5.58 µg of chloride ion per millilitre.

B-8.2.5 *Turbidity Meter Calibration* (*see* below informal Table)

B-8.2.5.1 Prepare a series of standards by taking 1 ml, 2 ml, 3 ml, 4 ml and 5 ml of standard chloride solution (1 ml equivalent to 5.58 µg/ml) in 100 ml graduated cylinders. Make up to 100 ml with water.

B-8.2.5.2 Add 1 ml of dilute nitric acid, mix well. Further add 1 ml of silver nitrate solution and mix carefully by inverting the cylinders. Do not shake the cylinders. Set these in the dark for about 15 min. Prepare a blank solution by taking 100 ml water and without chloride standard solution. Keep blank and standards in dark for 15 min. Measure the turbidity of the standards, by setting zero turbidity with blank solution. Plot the chloride concentration (ppm) versus turbidity reading to obtain a calibration curve. Calculate the slope.

B-8.2.6 *Ethylene Glycol Sample Analysis*

B-8.2.6.1 Take 50 ml glycol sample into a 100 ml graduated cylinder/nessler tube and add 50 ml of distilled water to cylinder. Take 50 ml of distilled water into a separate 100 ml graduated cylinder/ nessler tube for blank. Add 1 ml of dilute nitric acid (1 : 1) to both cylinders and mix well. Introduce 1 ml of 1.0 percent silver nitrate solution to each tube and mix well. Cap both the tubes and carefully invert the tube 2 to 3 times (do not shake). Set these in dark for 15 min. Set the turbidity instrument at zero point using blank and measure the turbidity of the sample.

Volume of Standard, ml (*1 ml = 5.58 µg / ml*)	*Equivalent Glycol Sample, ml*	*Chloride equivalent in Glycol*, ppm* (*w/w*)
1	50	0.1
2	50	0.2
3	50	0.3
4	50	0.4
5	50	0.5

*Chloride = Volume of standard x 5.58 µg/(50 ml x Glycol Specific Gravity)

NOTE — Consider Glycol Specific Gravity = 1.115 5

B-8.2.6.2 *Alternate method for ethylene glycol sample analysis for sample having chloride less than 0.2 ppm*

Take glycol sample and 0.2 ppm chloride standard in two separate graduated cylinder/Nessler tube. Develop turbidity by following all steps in clause **B-8.2.6.1** for both sample and 0.2 ppm chloride standard. Measure turbidity of glycol sample and 0.2 ppm chloride standard. If sample turbidity is less than 0.2 ppm chloride standard turbidity, report chloride in glycol sample less than 0.2 ppm. No calculation is needed.

B-8.2.7 *Calculation*

Calculate chloride in glycol corresponding sample turbidity reading from the above prepared calibration curve.

$$\text{Chloride (ppm, } w/w\text{), as Cl} = \frac{(A-B)}{S}$$

where

A = turbidity of sample, in NTU;

B = turbidity of blank, in NTU; and

S = slope of chlorides concentration vs turbidity NTU curve.

B-8.2.8 *Report*

Report the chloride to the nearest one decimal place as chloride, ppm.

B-8.3 Method B

B-8.3.1 *Outline of Method*

This method describes the determination of (inorganic) chloride in glycols in the range of 0.03 mg/kg to 10 mg/kg by potentiometric titration using standard silver nitrate solution. Chloride is determined by potentiometric titration with 0.001 N alcoholic silver nitrate standard solution in conjunction with a silver billet combination electrode. An automatic titrator or a *p*H meter in the millivolt mode may also be used to obtain the potentiometric titration end point.

B-8.3.2 *Apparatus*

B-8.3.2.1 *Potentiometric autotitrator*

Having measuring accuracy of ± 2 mV or better with silver indicating electrode equipped with automatic burette having 0.001 ml resolution.

B-8.3.2.2 *Magnetic stirrer with PTFE coated stirring bar*

B-8.3.2.3 *Analytical balance*, 0.1 mg readability

B-8.3.2.4 *Oven with accuracy,* ± 5 °C temperature

B-8.3.2.5 *Glasswares*, volumetric flasks, measuring cylinders, pipettes

B-8.3.3 *Reagents*

B-8.3.3.1 *Nitric acid* (*see* IS 264)

B-8.3.3.2 *Silver nitrate*

B-8.3.3.3 *Sodium chloride*

B-8.3.3.4 *Hydrochloric acid*

B-8.3.3.5 *Isopropyl alcohol*

B-8.3.3.6 *Acetone*

B-8.3.3.7 *Glacial acetic acid*

B-8.3.4 *Preparation of Standard Silver Nitrate Solution*

B-8.3.4.1 *Preparation of 0.1 N silver nitrate solution*

Grind about 25 g of crystalline silver nitrate and dry in oven at 105 °C ± 5 °C temperature for 2 h. Cool and store in dark coloured bottle.

Weight accurately 16.99 g ± 0.001 g silver nitrate and transfer it into 1 000 ml volumetric flask. Dissolve silver nitrate in 500 ml water and dilute with isopropyl alcohol up to mark. Mix well and store in amber coloured glass bottle.

B-8.3.4.2 *Standardization of 0.1 N silver nitrate*

Dry about 25 g of sodium chloride standard in oven at 105 °C ± 5 °C temperature for 2 h. Cool and store in bottle. Weigh accurately 0.15 g to 0.20 g dried sodium chloride in a conical flask and add 100 ml distilled water. Add about 1 ml potassium chromate indicator (5 percent in distilled water). Titrate with silver nitrate till reddish brown colour persists for 1 min. Record burette reading.

$$\text{Normality of silver nitrate (0.1 N)} = \frac{\text{Weight of sodium cholride (in g)} \times 100}{\text{Burette reading} \times \text{Molecular weight of sodium chloride (in g)}}$$

NOTE — Molecular weight of sodium chloride = 58.45 g.

B-8.3.4.3 *Preparation of 0.001 N standard silver nitrate solution*

Dilute 0.1 N standard silver nitrate solution 100 times with isopropyl alcohol.

Normality of silver nitrate (0.001 N) = (Normality of 0.1 N silver nitrate)/100

B-8.3.4.4 *Titration solvent preparation*

Mix 750 ml of acetone, 100 ml glacial acetic acid and 10 ml of 0.001 N hydrochloric acid (HCl). Store this solvent in cool and dry place and use after 24 h.

B-8.3.5 *Procedure*

B-8.3.5.1 *Blank determination*

Take 100 ml of a titration solvent in titration vessel and titrate with 0.001 N alcoholic silver nitrate standard solution. Record burette reading. Repeat blank measurement. In case if burette reading difference is more than 0.02 ml, repeat blank determination. Consider average of both blank as V_1.

B-8.3.5.2 *Sample analysis*

Weigh 50 g of glycol sample nearest to 0.01 g into the titration vessel. Add exact 100 ml titration solvent (using volumetric pipette) and mix well. Titrate with 0.001 N alcoholic silver nitrate solution. Perform this titration in duplicate and note average volume as V_2.

B-8.3.6 *Calculation*

Chloride content in glycol sample is calculated as:

$$\text{Chloride, mg/ kg} = \frac{(V_2 - V_1) \times 35.45 \times M \times 1\,000}{W}$$

where

V_2 = volume of silver nitrate for sample, in ml;

V_1 = volume of silver nitrate for blank, in ml;

M = normality of silver nitrate; and

W = mass of sample, in g.

B-8.3.7 *Report*

Report the chloride to the nearest one decimal place as chloride, ppm.

B-8.4 METHOD C

B-8.4.1 *Outline of Method*

This test method covers the determination of inorganic chloride in monoethylene glycol (MEG), diethylene glycol (DEG) and triethylene glycol (TEG) by ion chromatography.

A glycol sample is injected into an ion chromatograph having fixed sample loop, anion exchange columns (guard and separator) along with anion suppressor and conductivity detector. In this technique ions are separated according to its affinity towards the ion exchange resin and resins affinity for the eluent. Suppressor increases the conductivity of the analytes and decreases the conductivity of the eluent. Chloride is detected by change in conductivity and identified by retention time. Ion chromatograph is calibrated using external chloride standards and quantification in sample is done by response of chloride during sample analysis.

B-8.4.2 *Apparatus*

B-8.4.2.1 *Ion chromatograph*

B-8.4.2.1.1 Type A (Carbonate based Eluent system) or Type B (Hydroxide based Eluent system) with eluent pump, conductivity detector, PEEH tubing and six port injection valve or suitable sample injection system with fixed sample loop. An auxiliary regenerant pump or pressurized 4 litres reagent bottle.

B-8.4.2.1.2 *Guard column*

B-8.4.2.1.3 *Separator column*

B-8.4.2.1.4 *Anion suppressor*

B-8.4.2.1.5 *Chromatography data system*

B-8.4.2.2 *Analytical balance,* capable of weighing 220 g to the nearest 0.000 1 g

B-8.4.2.3 *Pipettes,* capable of measuring from 100 µl to 10 ml

B-8.4.2.4 *Filter,* 0.2 µm

B-8.4.2.5 *Plastic syringes,* 5 ml and 10 ml

B-8.4.2.6 *Volumetric flasks,* 100 ml

B-8.4.2.7 *Plastic cups*

B-8.4.3 *Reagent*

B-8.4.3.1 *Sodium carbonate* (Na_2CO_3), 0.5 M

B-8.4.3.2 *Sodium bicarbonate* ($NaHCO_3$), 0.5 M

B-8.4.3.3 *Sodium chloride* ($NaCl$) or *certified reference material (CRM)*, of 1 000 ppm chloride

B-8.4.3.4 *Sodium hydroxide* ($NaOH$), 50 percent

B-8.4.4 *Instrument Conditions*

B-8.4.4.1 *Type A: ion chromatography — carbonate based eluent system*

Columns	:	Carbonate selective anion exchange guard column 4 × 50 mm and separator column 4 × 250 mm
Injector	:	PEEK 6 port valve
Detector	:	Conductivity Cell at 35 °C
Suppressor and Mode	:	Chemical or electrolytic anion suppressor at 50 mA. Deionized or external water regenerant at 4 ml/min.
Column Temperature	:	30 °C
Eluent	:	0.6 mM* Sodium carbonate (Na_2CO_3)/0.4 mM Sodium bicarbonate ($NaHCO_3$) *mM is millimolar
Eluent Flow Rate	:	2 ml/min
Sample Loop	:	70 µl
System Pressure	:	9.8250 MPa (1425 psig) or optimal pressure
Runtime	:	5 min or optimal run time
Background conductance	:	10 µS or optimal (µS is micro siemens)
Retention time	:	Approximately 2.3 min for chloride

B-8.4.4.2 *Type B ion chromatography — hydroxide based eluent system*

Columns	:	Hydroxide selective anion exchange guard column 4 × 50 mm and separator column 4 × 250 mm
Injector	:	PEEK 6 port valve
Detector	:	Conductivity cell at 35 °C
Suppressor and Mode	:	Chemical or electrolytic anion suppressor at 50mA. Deionized or external water regenerant at 4 ml/min.
Column Temperature	:	30 °C
Eluent	:	5 mM* Potassium hydroxide (KOH) *mM is millimolar.
Eluent Flow Rate	:	1 ml/min.
Sample Loop	:	70 µl
System Pressure	:	9.8250 MPa (1425 psig) or optimal pressure
Runtime	:	5 min or optimal run time
Background conductance	:	10 µS or optimal (µS is micro siemens)
Retention time	:	approximately 2.3 min for chloride.

B-8.4.5 *Instrument Calibration*

B-8.4.5.1 Prepare a stock solution of 1 000 mg/kg chloride in water using sodium chloride or alternatively use 1000 µg/ml chloride standard. Prepare 10 mg/kg stock solution by diluting 1 000 mg/kg chloride standard 100 times with water. Prepare chloride working standard solution of 0.01 mg/kg, 0.02 mg/kg, 0.05 mg/kg, 0.1 mg/kg, 0.2 mg/kg, 0.5 mg/kg and 1.0 mg/kg in glycol by the following:

Chloride Working Standard	*Nominal Concentration*	*Weight of 10 mg/kg Chloride Calibration Solution* (*to add*)	*Final Weight of Chloride Working Standard* (*MEG, DEG or TEG added*), *g* (± *0.001 g*)
(2)	(3)	(4)	(5)
1	0.00	0	100
2	0.01	0.1	100
3	0.02	0.2	100
4	0.05	0.5	100
5	0.1	1.0	100
6	0.2	2.0	100
7	0.5	5.0	100
8	1.0	10.0	100

NOTE — Use high purity glycol for dilution.

B-8.4.5.2 Turn ON the Ion Chromatograph and allow to equilibrate until baseline and background conductance are stable. Take two - three blank run of the glycol from which standards are prepared to nullify chloride from final results. Inject and run chloride standards in duplicate/triplicate into instrument for calibration. If auto sampler is available, then put the sample in vial else for manual injection use plastic syringe. Identify the chloride peak by its retention time. A calibration curve can be prepared by instrument software or manually by plotting chloride standards peak area v/s chloride standard concentrations.

B-8.4.5.3 Calculate slope using the following formula

$$y = mx + c$$

where

y = peak area of calibration standards;

m = slope of linear line; and

x = concentration of calibration standards;

c = intercept

NOTE — $c = 0$, if blank area is deducted from standard area, then curve is passing through zero point without intercept.

B-8.4.5.4 Evaluate the linearity of the calibration by calculating correlation coefficient (r) and shall be greater than equal to 0.99. To verify the calibration, a quality control check standard of chloride is to be analysed.

B-8.4.6 *Glycol Sample Analysis*

B-8.4.6.1 Glycol samples may be analysed with or without dilution. Glycol sample may be diluted with chloride free distilled water for chloride analysis in the desired ratio, preferably 50 : 50 or another suitable ratio, in case of dilution. Inject glycol sample into ion chromatograph and either calculate result manually or with instrument software.

B-8.4.7 *Calculation*

B-8.4.7.1 Chloride in glycol sample, when linear calibration curve passed through zero.

$$\text{Concentration} = \frac{Peak\ Area}{Slope\ of\ the\ linear\ Regression}$$

B-8.4.7.2 *Chloride in glycol sample*, when linear calibration curve is not forced to zero.

B-8.4.8 *Report*

Report the chloride to the nearest one decimal place as chloride, ppm

B-8.4.9 *Precision*

B-8.4.9.1 *Repeatability*

Repeatability of chloride in glycol is 0.01 mg/kg at 0.078 mg/kg standard.

B-8.4.9.2 *Reproducibility*

Reproducibility has not been evaluated due to insufficient analysis data.

B-9 DETERMINATION OF ETHYLENE GLYCOL (PURITY) AND DIETHYLENE GLYCOL

B-9.1 General

This test method is used to determine glycol purity and impurity in ethylene glycol in the range of 22 mg/kg to 3 000 mg/kg. A small volume of sample is injected into a gas chromatograph and passes through capillary column, where the separation take place and FID is used for detection. The concentration of each impurity is calculated by external standard method.

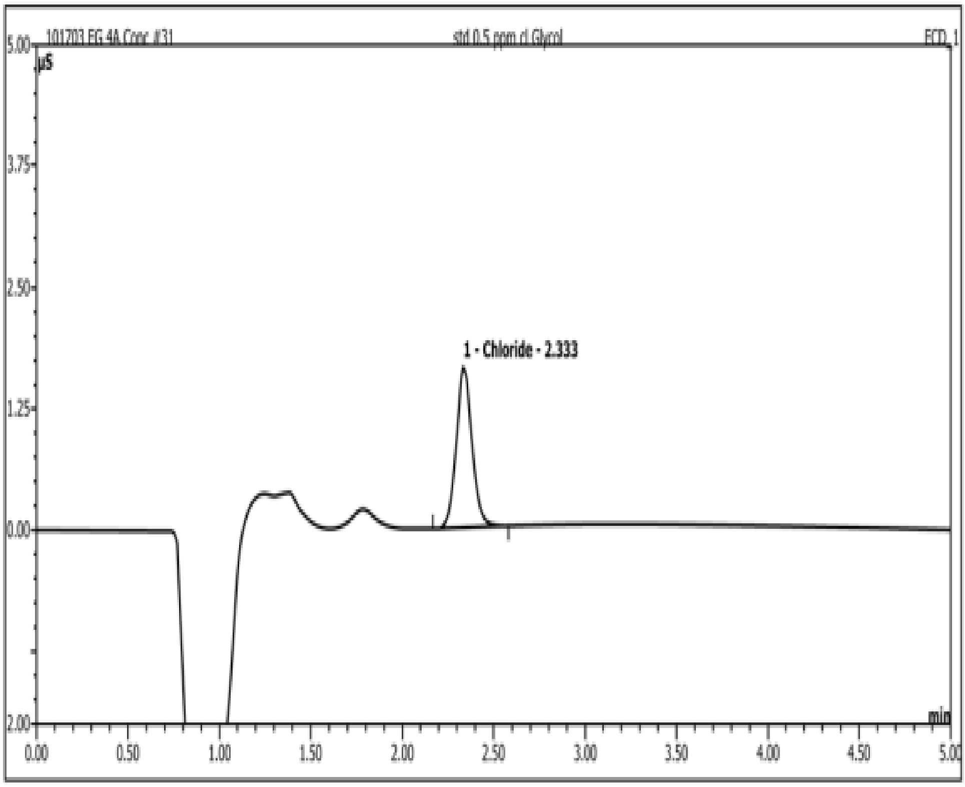


FIG. 1 CHROMATOGRAM OBTAINED FROM TYPE A IC (CARBONATE BASED ELUENT SYSTEM)

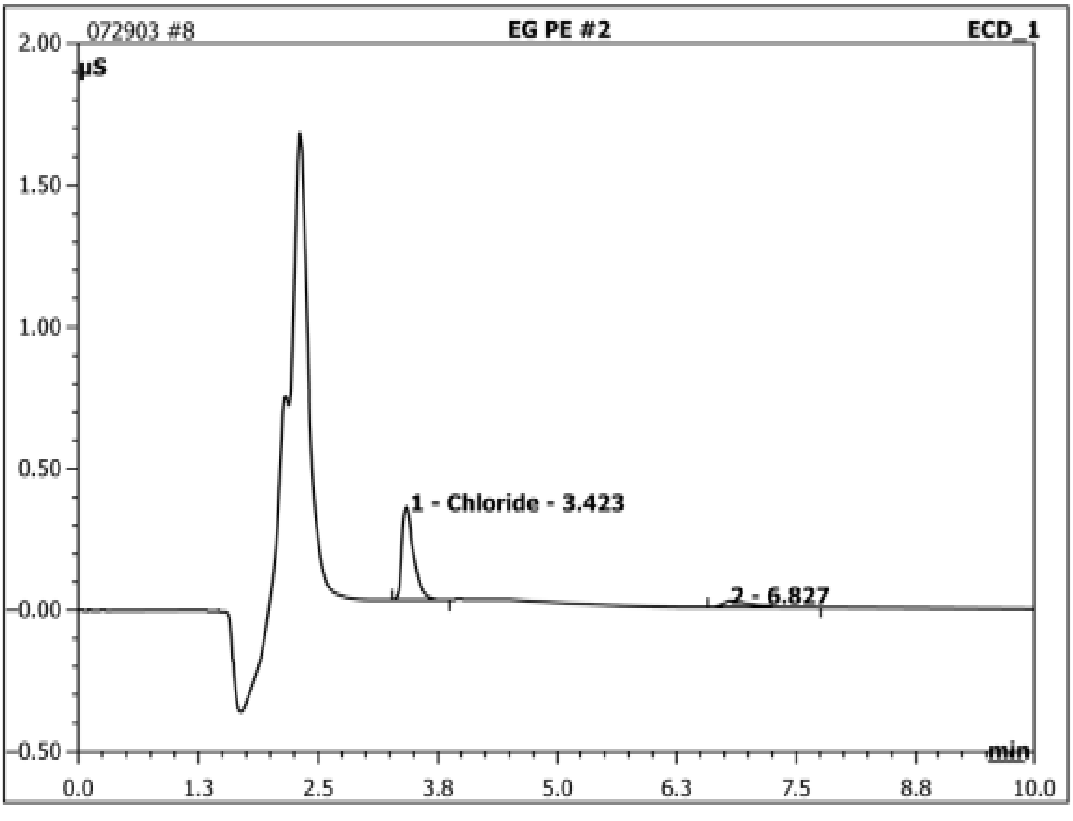


FIG. 2 CHROMATOGRAM OBTAINED FROM TYPE B IC (HYDROXIDE BASED ELUENT SYSTEM)

B-9.2 Apparatus

B-9.2.1 *Gas Chromatograph*

Any gas chromatograph equipped with flame ionization detector (FID), a split/splitless injector and a suitable electronic integrator/software may be used with following accessories and operating condition.

B-9.2.1.1 *Column,* capillary wide-bore, fused silica column with a stationary phase of polyethylene glycol, length 15 m; internal diameter 0.53 mm and film thickness 1 µm or equivalent.

B-9.2.1.2 *Gas chromatographic condition parameters*

Injector

a) Injection volume : 0.5 µl to 1 µl using split injection technique

b) Split ratio : 1 : 10 or appropriate split ratio to allow adequate sensitivity

Oven Temperature

a) Start : 70 °C for 0.05 min

b) Ramping : 25 °C/min from 70 °C to 230 °C

c) End : 230 °C for 10 min

Detector :

a) Temperature : 250 °C

Carrier Gas : Helium and Nitrogen

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness etc), calibration technique (internal standard, external standard, area normalization, percent area etc), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

B-9.2.2 *Analytical Balance*, minimum capacity 220 g capable of weighing to the nearest 0.1 mg.

B-9.2.3 *Syringe*, 10 µl

B-9.3 Reagents

B-9.3.1 *Monoethylene Glycol*, 99 percent purity, minimum

B-9.3.2 *Diethylene Glycol*, 99 percent purity, minimum

B-9.4 Calibration

B-9.4.1 Prepare at least three calibration solutions containing 200 mg/kg, 500 mg/kg and 1 000 mg/kg of each of the glycol components to be determined, by adding the relevant standard to a blank sample of ethylene glycol and mix thoroughly. Analyze each calibration solution and the blank solution at least three times using the operating parameters

$$C_i = \frac{W_i}{W_i + W_{blank}}$$

where

C_i = concentration of each glycol component in the calibration standard, in g;

W_i = mass of glycol component added to the calibration standard, in g; and

W = mass of glycol component added to the calibration standard, in g.

B-9.4.2 Prepare a plot of area counts of the glycol component (*y*-axis) versus the concentration of the glycol component (mg/kg) added to the calibration solution (*x*-axis). Using a computer program, determine the best-fit line through the data using linear regression analysis. The relationship between concentration and peak area will be linear. Record the intercept value (concentration; mg/kg) where the resulting line crosses the *x*-axis ($y = 0$). Apply digital integration equipment for measuring the peak areas. Calculate the corrected concentration (mg/kg) of each of the glycol component in each calibration solution by using the following formula:

$$\text{Corrected } C_i = C_i + \text{Y}$$

where

Corrected C_i = Corrected concentration (mg/kg) of the glycol component in each calibration standard;

C_i = the concentration (mg/kg) of the glycol component added to the calibration standard; and

Y = absolute value of the concentration of blank glycol determined from the linear regression graph (intercept value) for each calibration standard.

B-9.4.3 For each chromatogram, calculate the system response factor *(f)* of each of the glycol components by means of the following equation:

$$f = \frac{\mathit{Corrected}\ C_i}{A_i}$$

where

Corrected C_i = corrected concentration (mg/kg) of the glycol component in each calibration standard; and

A_i = peak area of component

Calculate the mean of the response factors for each of the glycol components.

NOTE — GC software may also plot calibration graph for the component of interest automatically.

B-9.5 Procedure

Ensure that GC is set as recommended before injecting the sample. Inject the sample in GC once it attains room temperature.

B-9.6 Calculation

Obtain the concentrations of the ethylene glycol and its impurities in the test sample in mg/kg, as presented by the software of the applied gas chromatograph. If an automated system is not being applied then calculate the concentration of the glycol components, in mg/kg, in the test sample by means of the following equation.

$$\text{Glycol components} = A_i \times f$$

where

A_i = peak area of relevant glycol component; and

f = average response factor of component.

Purity of ethylene glycol, weight percent =100 − (other glycol impurities + water content of the sample)

NOTE — For water content refer Sl No. (iv) of Table 1.

B-10 DETERMINATION OF ALDEHYDE (*as* HCHO)

B-10.1 General

This test method describes the spectrophotometric determination of total aldehyde content in the range of 0.5 mg/kg to 50 mg/kg (as acetaldehyde) or 0.3 mg/kg to 35 mg/kg (as formaldehyde) in mono ethylene glycol. The sample is treated with 3-methyl-2-benzothiazone hydrazone hydrochloride (MBTH) which reacts quantitatively with free aldehyde to form the azine derivative and water. The excess MBTH is oxidize with acidic ferric chloride to a reactive cation which then forms a blue green complex with the azine, whose colour intensity corresponds to the aldehyde content and is measured photo metrically at 635 nm.

B-10.2 Apparatus

B-10.2.1 *UV-Visible Spectrophotometer*

B-10.2.2 *Analytical Balance***,** capable of weighing 200 g to the nearest 0.000 1 g

B-10.2.3 *Glass Absorber with Connecting Tube*, two of capacity 250 ml

B-10.2.4 *Stopwatch*

B-10.2.5 *Volumetric Flask*, 50 ml and 100 ml

B-10.2.6 *Pipettes*, 1 ml, 2 ml, 5 ml, 10 ml and 20 ml

B-10.2.7 *Cuvette*, 10 mm

B-10.2.8 *Rotameter*, for gas flow measurement

B-10.3 Reagents

B-10.3.1 *3-Methyl-2-benzothiazone Hydrazone Hydrochloride* (*MBTH*)*,* 0.4 percent solution in distilled water

B-10.3.2 *Ferric Chloride,* 0.3 percent solution in distilled water

B-10.3.3 *Formaldehyde,* AR Grade, 36 percent to 40 percent

B-10.3.4 *Methanol or Acetone*

B-10.3.5 *Hydrochloric Acid*

B-10.4 Calibration

B-10.4.1 *Standard Formaldehyde Solution*

Weigh 2.0 g to 2.2 g of formaldehyde and transfer it to 100 ml volumetric flask. Make it up to the mark with water and mix thoroughly. This is stock solution (A) having 1.0 ml = 1 000 µg formaldehyde (HCHO). Now prepare 4 µg/ml working solution by serial dilution of 1 ml above stock solution to 250 ml in water.

Now prepare a series of standards as per the following table in 100 ml volumetric flask.

Volume of Aldehyde Standard; 1 ml = 4 µg	*Concentration, µg*
0 (blank)	0
1	4
2	8
3	12
4	16
5	20
6	24
7	28
8	32

B-10.4.2 *Colour Development*

Add 5 ml MBTH solution by volumetric pipette to each standard solution and blank as prepared in **B-10.4.1**. Thoroughly swirl the flasks and loosely stopper the flask. Allow to stand for 30 min in dark. After 30 min, add 25 ml ferric chloride solution and dilute it to 100 ml with methanol or acetone. Mix the solution properly. Now measure the absorption of the test solution at 635 nm using 10 mm cell. To check the aldehyde concentration follow instrument manufacturer directions.

B-10.5 Procedure

Take 1.0 ml of sample in 100 ml volumetric flask. To it add 5 ml MBTH and 4 ml water. Prepare a blank in same way by taking 5 ml of water and 5 ml of MBTH. Allow the flasks to stand for 30 min. After 30 min, add 25.0 ml ferric chloride solution and make it up to 100 ml with methanol. Mix the solution properly and allow it to stand for 5 min. After 5 min take absorption at 635 nm using 10 mm cell and find out Aldehyde concentration from calibration graph. To check the aldehyde concentration follow instrument manufacturer directions

B-10.6 Calculation

$$\text{Aldehyde, mg/kg} = \frac{\text{Aldehyde from graph}}{\text{Volume of sample x Density}}$$

B-10.7 Report

Report the result in ppm.

B-10.8 Precision

Sample type	*Aldehyde, mg/kg*	*Repeat-ability*	*Reproducibility*
Methylene Glycol	4.22	0.35	3.63

ANNEX C

(*Clause* 7)

SAMPLING OF ETHYLENE GLYCOL

C-l GENERAL REQUIREMENTS

C-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed,

C-l.2 Samples shall not be taken in an exposed place.

C-l.3 The sampling instrument shall be clean and dry and shall be made of low or reduced spark generating material.

C-l.4 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination (*see* also **4.1**).

C-l.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling, so as to bring all portions into uniform distribution.

C-l.6 The samples shall be placed in suitable, clean, dry and airtight glass containers preferably of amber or blue colour.

C-l.7 The sample containers shall be of such a size that they are almost but not completely, filled by the sample.

C-l.8 Rubber stoppers or composition corks shall not be used for closing the sample bottle.

C-1.9 Sealing wax or other plastic material, if used, shall be applied in such a way that it does not contaminate the sample when the bottles are opened.

C-1.10 Each sample container shall be protected by covers of oil-proof paper, metal foil, viscose or other suitable impervious material over the stopper to keep away moisture and dust from the mouth of the bottle and to protect it while being handled.

C-1.11 Each sample container shall be sealed airtight with a suitable stopper after filling and marked with full details of sampling, such as the date of sampling, the year of manufacture of the material, the batch number, the name of the supplier, etc. Particular care shall be taken to ensure that sealing methods do not contaminate the sample.

C-2 SAMPLING INSTRUMENT

C-2.1 The following forms of sampling instruments may be used:

a) Sampling bottle or can, for taking samples from various depths of large tanks; and
b) Sampling tube.

C-2.1.1 *Sampling Bottle or Can*

It consists of weighed bottle or metal container with removable stopper or top, to which is attached a light chain (*see* Fig. 3). The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the topper or top is removed by means of the chain for filling the container.

C-2.1.2 *Sampling Tube*

It is made of metal or thick glass and is about 20 mm to 40 mm in diameter and 400 mm to 800 mm in length (*see* Fig. 4). The ends are conical and reach 5 mm to 10 mm diameter at the tips. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

C-2.1.3 *For small containers*, the size of the sampling tube may be altered suitably.

C-3 SCALE OF SAMPLING

C-3.1 Lot

In any consignment, all the containers of the same size and drawn from same batch of manufacture shall constitute a lot.

C-3.2 Tests shall be conducted on each lot separately for ascertaining its conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with co1 (2) and col (3) of Table 2.

Table 2 Scale of Sampling

(*Clause* C-3.2)

Sl No. (1)	**Lot Size** (*N*) (2)	**Number of Containers to be Selected** (*n*) (3)
i)	Up to 25	3
ii)	26 to 50	4
iii)	51 to 100	5
iv)	101 to 300	6
v)	301 to 500	7
vi)	501 and over	8

NOTES

1 In case of large-sized containers, such as tanks, tank lorries or tankers, every container in the lot shall be sampled separately.

2 In case of very small lots where the selection of three containers may be uneconomical, the number of containers to be selected and the method of judging the conformity of the lot to the requirements of this specification shall be as agreed to between the purchaser and the supplier.

C-3.3 The containers shall be chosen at random from the lot with the help of a suitable random number table. In case no such table is available the following procedure is recommended for use.

Starting from any container of the lot, count them as 1, 2, 4... up to r and so on in one order, where r is the integral part of *N*/n (*N* being the number of containers in the lot and *n* the number of containers to be selected). Every r^{th} container thus counted shall be withdrawn to give sample for tests.

C-4 PREPARATION OF TEST SAMPLES

C-4.1 From each of the containers selected according to **C-3.2** and **C-3.3**, small portions of the material shall be drawn with the help of the suitable sampling instrument (*see* **C-2**) from several positions at the upper, middle and bottom layers according to Table 3. These portions shall be mixed together so as to yield a quantity of material not less than 1 000 ml representative of that container. From every large-sized container, a quantity of material not less than 1 000 ml representative of the container shall be made up of portions drawn according to Table 3.

C-4.2 From each of these individual samples, an equal quantity of material shall be taken and mixed thoroughly to form a composite sample, of about 1 000 ml. The composite test sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

C-4.3 The parts of the composite test sample, obtained according to **C-4.2**, shall be transferred to separate containers and shall be sealed and marked with full identification particulars given in **C-1.11**.

C-4.4 The reference test sample shall bear the seals of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier to be used in the case of any dispute between the two.

C-5 CRITERIA FOR CONFORMITY

C-5.1 The lot shall be declared as conforming to the requirements of this specification if the composite sample does not fail to satisfy any of the specified requirements.

Table 3 Depths of Sampling Layer and Proportions of the Sample

(*Clause* C-4.1)

Sl No.	**Depth of Content (percent) of the Diameter**	**Depths of Sampling Layer (Height from Bottom, percent of the Diameter)**			**Proportions of the Sample to be Drawn**		
		Upper Layer	Middle Layer	Bottom Layer	Upper Layer	Middle Layer	Bottom Layer
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	100	80	50	20	3	4	3
ii)	90	75	50	20	3	4	3
iii)	80	70	50	20	2	5	3
iv)	70	—	50	20	—	6	4
v)	60	—	50	20	—	5	5
vi)	50	—	40	20	—	4	6
vii)	40	—	—	20	—	—	10
viii)	30	—	—	15	—	—	10
ix)	20	—	—	10	—	—	10
x)	10	—	—	5	—	—	10

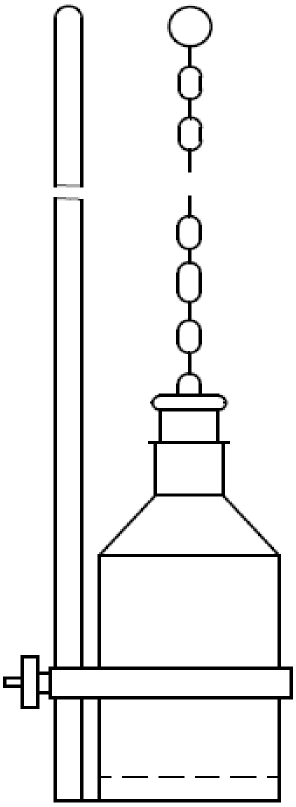
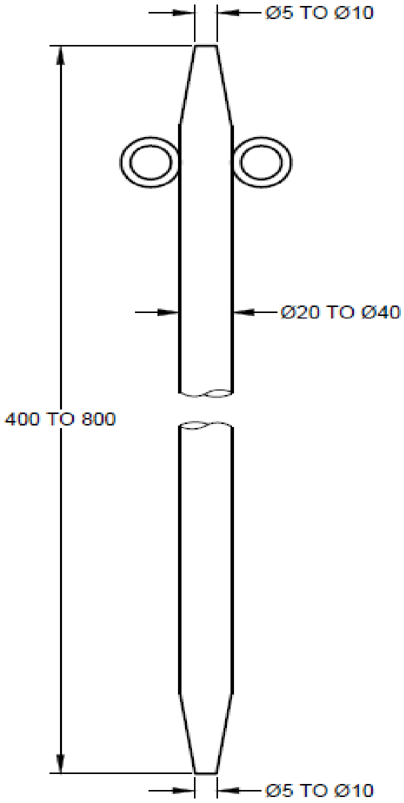


FIG.3 SAMPLE BOTTLE OR CAN



All dimensions in millimeters.
FIG. 4 SAMPLING TUBE

ANNEX D

(*Foreword*)

COMMITTEE COMPOSITION

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

Organization	*Representative(s)*
National Chemical Laboratory (NCL), Pune	Dr C. V. Rode **(*Chairperson*)**
Alkyl Amines Chemicals Limited, Mumbai	Shri S. V. Nikumbhe Shri Sameer Katdare (*Alternate*)
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	Shri K. L. Raphael Shri Kirti Gajjar (*Alternate*)
All India Distillers Association (AIDA), New Delhi	Shri V. N. Raina
BASF India Limited, Mumbai	Shri Kiran Bhat Shri Hemal (*Alternate*)
CSIR-Central Drug Research Institute (CDRI), Lucknow	Dr Sanjeev Kanojiya
Chemical and Petrochemicals Manufacturers Association (CPMA), New Delhi	Shri Uday Chand
Deepak Fertilizer, New Delhi	Dr L. B. Yadawa Shri Suresh Amle (*Alternate*)
Deepak Phenolics Limited, Vadodara	Shri Dharmesh Siddhapuria Shri Sandip Kumar Pandya (*Alternate*)
Department of Chemicals and Petrochemicals, Ministry of Chemicals and Fertilizers, New Delhi	Shri O. P. Sharma Shri Varun Singh Poonia (*Alternate*)
DOW Chemical International Private Limited, Mumbai	Shri V. Mohandoss Shri Govind Gupta (*Alternate*)
Godavari Biorefineries, Mumbai	Shri Shanul Laxmanrao Pagar Shri Appasaheb J. Wani (*Alternate*)
Gujarat Narmada Valley Fertilizers Company Limited, Ahmedabad	Dr R. M. Patel Shri C. S. Patel (*Alternate*)
Hindustan Organic Chemicals Limited (HOCL), Mumbai	Shri Deleep Kumar K. Shri Pradeesh Narayanan (*Alternate*)
INEOS Styrolution India Limited, Vadodara	Dr Kanak Dass
India Glycols Limited, Kashipur	Dr R. K. Sharma Shri Alok Singhal (*Alternate*)
Indian Chemical Council (ICC), Mumbai	Dr Mritunjay Chaubey Shri J. Sevak (*Alternate*)

Organization	*Representative(s)*
Indian Oil Corporation Limited, Panipat	DR Y. S. JHALA
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This Indian Standard has been developed from Doc No.: PGD 09 (21295).

Amendments Issued Since Publication

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Published by BIS, New Delhi